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(54) Method for dry fractionation of fatty substances.

(57) The present invention relates to a method for dry fractionating oils and fats comprising a nucleation stage, a crystal growth stage and a solids separation stage, wherein at least during the crystal growth stage a triglyceride oil or fat of a composition other than the composition of the oil or fat being dry fractionated is added to the oil or fat being dry fractionated.

EP 0 651 046 A1

Background of the invention

The present invention relates to a method for dry fractionating oils and fats comprising a nucleation stage, a crystal growth stage and a solids separation stage, wherein at least during the crystal growth stage a triglyceride oil or fat of a composition other than the composition of the oil or fat being dry fractionated is added to the oil or fat being dry fractionated.

Natural glyceride oils and fats comprise many different triglycerides, the physical properties of which to a large extent are determined by chain lengths and degree of unsaturation of the fatty acid moieties. To make natural glyceride oils and fats more suitable for particular applications it is often required to separate them into fractions which are more homogenous with respect to melting behaviour.

It is known to fractionate oils and fats in the presence of an organic solvent such as acetone or hexane. Although this method of fractionation is highly efficient, it is not preferred because flammable solvents are used and because it is expensive.

It is also known to fractionate oils and fats by using an aqueous solution of a surface active agent. However, this method has the disadvantage of low fractionation efficiency in view of yield and quality. Besides, post-treatment for separating the aqueous solution from the oil or fat is complicated and high costs are involved for treating surfactant containing waste water.

Another method is the dry fractionation process which is carried out in the absence of classical organic solvents. This method comprises forming crystal nuclei (nucleation stage) by cooling the molten oil or fat to below its melting point, ageing them at a low temperature to allow the crystals to grow (crystal growth stage) and finaly subjecting the suspension to solid-liquid separation (solids separation stage). This latter fractionation method is the cheapest and simpliest method known in the art, but it has certain inherent disadvantages. The first disadvantage is that during the separation stage, when the partially crystallized melt is separated into a stearin fraction and an olein fraction, the crystals in the stearin fraction entrain an amount of liquid. Since this liquid is undiluted olein as opposed to an olein solution as is the case in solvent fractionation, its relative amount is rather high leading to a less selective fractionation with respect to the stearin fraction and a lower olein yield in comparison with solvent fractionation.

A second disadvantage is that the solids content of the crystallizer is limited to what the crystallizer agitator can keep in suspension and what the pump feeding the filter in the separation stage can handle. In comparison with the solvent fractionation process, the dry fractionation process can therefore isolate relatively less solid material and if more solid material has to be removed e.g. to attain a certain olein quality, the dry fractionation process has to resort to operate in successive stages which increases its cost.

The third disadvantage of the dry fractionation process is that during a crystallization crystals having different morphology may be formed. At the early stages, fairly large and hard crystals may for instance be formed whereas at a later stage much smaller crystals may emerge. These smaller crystals may adversily affect filtration characteristics and lead to a filter cake with a relatively low content of crystalline material and thus cause a deterioration of the selectivity of the fractionation.

Therefore, a number of process modifications have been proposed to increase fractionation selectivity and improve filterability of the crystallized fat.

One such process modification has been proposed in US 4,265,826. In this method, the oil or fat to be dry fractionated is seeded, at a temperature above its slip melting point, with seed crystals of high melting point components of said oil or fat and subsequently cooled to below its slip melting point to crystallize the high melting point components. Seeding the oil to be dry fractionated with high melting point components is said to shorten the crystal growth stage and to improve filtration efficiency. Although this process modification has been reported in the examples given to be profitable, it has been found not to overcome the third disadvantage quoted above.

Another similar process modification has been proposed in EP-A 188 015, which modification comprises admixing with the oil or fat to be dry fractionated, a second triglyceride oil containing tripalmitin and lowering the temperature of the admixture from a temperature at which it is fully molten to a temperature at which the stearin fraction crystallises. This process modification is said to avoid the formation of fat crystals which hamper filtration. However, we have observed that when crystallization is continued and the cooled admixture is depleted in tripalmitin, small crystals which adversily affect filtration are formed anyway. However, we have observed, as for US 4,265,826, that smaller crystals which adversily affect filtration are formed when crystallization is continued and the cooled admixture is depleted in tripalmitin.

In EP-A 256 760 a method is disclosed for filtering a suspension comprising fat crystals of a paste-like consistency. This method comprises introducing the fatty material in a fluid state into a filter chamber of a filter apparatus having pressing means, maintaining the fatty material therein at crystallization conditions to crystallize the higher melting compounds and actuating the pressing means to squeeze out the liquid oil.

The filter chamber is initially coated with a fat layer, solidified by cooling this layer to far below its melting point, to prevent the liquid oil to be dry fractionated to percolate through the filter cloth before the crystallisation stage is completed. Although this method brings about an effective separation of the liquid fraction from the higher melting point components, it is hardly used industrially because it is expensive. As a matter of fact, crystallization of the higher melting point components takes some considerable time as a result of which throughput of the installation, which is expensive, is rather low.

Another modification of the dry fractionation method is disclosed in EP-A 0 249 282 and is designed for the dry fractionation of fat blends which contain a considerable amount of crystallized fat at the fractionation temperature applied. Such fats are not pumpable and separation of the fractions is almost impossible owing to the rheological properties of the mass. This process modification comprises blending the fat to be dry fractionated with an oil which is substantially liquid at low temperatures, heating the blend to a temperature above the melting point, cooling the mixture and finally separating the higher melting fraction from a mixture of the lower melting fraction and the liquid oil.

However, throughput capacity of the installation is seriously reduced. As a matter of fact, a considerable amount of fat to be dry fractionated is replaced by liquid oil, which serves the purpose of diluting the suspension. In addition, the liquid oil added in accordance with this process further affects stearin quality.

Another method to increase the efficiency and yield of the dry fractionation is disclosed in EP-A 399 597. According to this method the olein fraction obtained after dry fractionation of the starting fatty material and separation by membrane filter pressing is subjected to a similar dry fractionation at a lower crystallization temperature and the stearin fraction obtained is recycled to the fatty material to be dry fractioned in the first dry fractionation treatment. This method allows an effective separation between the higher melting point components and the lower melting point components, but it requires more time and effort to achieve the fractionation.

Object of the invention

It is the object of the present invention to provide an improved method for fractionating oils and fats, which method approximates the efficiency of solvent fractionation and which is as simple and cheap as the dry fractionation method.

Detailed description of the invention

According to the present invention, there is provided a method for dry fractionating oils and fats comprising a nucleation stage, a crystal growth stage and a solids separation stage, wherein at least during the crystal growth stage a triglyceride oil or fat of a composition other than the composition of the oil or fat being dry fractionated is added to the oil or fat being dry fractionated.

As set out earlier, addition of high melting point components as seeding crystals to the oil or fat to be dry fractionated does not avoid the subsequent formation of a paste-like suspension of very fine crystals. We have now found that, by addition of a fat comprising triglycerides which easily crystallize out at fractionation temperature to the oil or fat being dry fractionated, during the crystal growth stage, the formation of a paste-like suspension is avoided.

By crystal growth stage is understood the stage at which the oil or fat being dry fractionated is an apparent suspension of very fine crystals in liquid oil. As a rule, the crystal growth stage is meant to refer to the stage at which the oil or fat being dry fractionated has a solid fat content of at least 1 %. It is however obvious that during this stage new nuclei may be formed.

Why and how the formation of this paste-like consistency is avoided when applying the method according to the present invention is not clear but we have the impression that it is connected with the formation of mixed crystals that above a certain content of higher melting triglycerides grow in a form that leads to good filterability but below this content grow as very small crystals that impede filtration. Whereas in processes according to the state of the art the crystal growth stage has to be stopped by starting the separation stage some time before the liquid phase has become so far depleted with respect to higher melting triglycerides that crystals with poor filterability are formed, the addition of said higher melting triglycerides during the crystal growth stage according to the present invention allows this stage to be continued.

As a result, a higher solids content in the crystallizer prior to filtration can be attained while maintaining filterability leading to a higher stearin yield and an olein with a higher iodine value. In fact, the process according to the invention can in some instances achieve in a single step where the state of the art methods require two steps.

The amount of oil or fat added to the oil being dry fractionated and its rate of addition may be determined experimentally. They depend strongly upon the oil or fat being fractionated but we have also found that they can be varied between rather wide limits without seriously affecting the filtration characteristics and olein properties. In general, it has been found advantageous to add the triglyceride oil or fat having a composition other than the oil or fat being dry fractionated gradually and over an extended period of time. It has been found that adding more during the early part of the crystal growth stage hardly diminishes the amount that has to be added during later parts of the crystallisation stage. This means that if the triglycerides added during the crystal growth stage cause the stearin properties to deviate from target, the start of the addition of oil or fat of a composition other than the oil or fat being dry fractionated is preferably postponed as much as possible.

The triglyceride oil or fat having a composition other than the oil or fat being dry fractionated is preferably added as a molten liquid oil because this is more easy to handle than a crystal slurry. If then temperature is above the temperature maintained in the crystallizer, a slow and gradual addition into the agitated crystallizer will not seriously affect its temperature control. If, on the other hand, a stepwise addition is preferred and temperature control in the crystallizer might be hampered if the temperature difference is too large, this difference can be lowered by diluting/dissolving the oil or fat having a composition other than the oil or fat being dry fractionated with an oil that is substantially liquid at crystallisation temperature. Cooling the oil or fat to crystallisation temperature just prior to addition is another possibility.

In the state of the art method described in EP-A 0 249 282, a fat (A) that has a high solid fat content at the crystallisation temperature is blended with an oil (B) that is substantially liquid at this temperature and then fractionated as a blend. This method thus allows a higher proportion of the fat (A) to be crystallized while maintaining proper agitation but necessarily leads to inclusion of the oil (B) in the stearin filter cake and thus alters stearin properties in a possibly undesirable direction.

The process according to the invention can effectively overcome this drawback if the oil being added during the crystal growth stage is the olein fraction resulting from the ongoing fractionation process. In this embodiment of the present invention the fat or oil to be fractionated is allowed to crystallize to such an extent that it can still be agitated, at which point in time filtration is started and stearin and olein are collected, whereby the olein is recycled to the crystallizer as to dilute the crystal slurry. Crystallization is allowed to continue until the olein has the properties aimed for, at which point in time the olein resulting from the separation stage is collected and no longer recirculated.

In another, particularly useful embodiment of the invention the addition of a fat containing higher melting triglycerides and recycling of the olein isolated from the ongoing fractionation are combined. If then the rate of stearin removal is about the same as the rate of addition of the fat containing the higher melting triglycerides, the volume of the batch being processed hardly changes, leading to maximum crystallizer utilisation. In addition, a single step process achieves what otherwise requires several steps.

The combination indicated above can be effectively applied in the fractionation of palm oil leading to a single step, high yield production of high iodine value palm olein. In this process, palm oil is crystallized at a temperature of for instance 17 °C in a crystallizer that is filled with palm oil. When the solids content of the crystallizer reaches for example 10 %, filtration is started, the resulting olein is recycled and at the same time, palm oil is slowly added to the crystallizer, either as a liquid at a temperature just above its melting point or as a mixture with the recycled palm olein which may then have a lower temperature. Recycling of palm olein and addition of palm oil are continued until an iodine value of 63-65 or even higher for the olein is observed but in practice, good results have also been obtained when the addition of palm oil was stopped when the olein had an iodine value of 60. Because filterability of the crystals remains good until the very last moment, olein with a high iodine value can thus be produced in a single step at 60 % yield based upon the total palm oil input.

Suitable oils or fats which may be fractionated according to the process of the present invention include animal oils or fats such as lard, tallow and whale oil, and vegetable oils or fats such as soybean oil, rapeseed oil, peanut oil, cottonseed oil, rice oil, corn oil and palm oil. It should be understood that the foregoing list of oils or fats is intended to embrace the hydrogenated and/or ester-interchanged forms and fractions thereof.

The present invention is now illustrated by the following examples.

Example 1

In this example a 7 l laboratory crystallizer was used. It consisted of a cylindrical glass vessel provided with a water jacket connected to a thermostat that can heat as well as cool, and with a helical agitator. An amount of 3075 g palm olein was introduced into the vessel, heated to a temperature of 60 °C, kept at this

temperature for 1 hour and then cooled to a temperature of 16 °C. When crystallisation was observed to start, a mixture of 1300 g of the same palm olein and 625 g palm oil, that was maintained at a temperature of 25-30 °C and therefore remained liquid was added to the crystallizer by means of a peristaltic pump at a rate of addition of on average 76 g/hour. When the total amount of this mixture had been added after some 25 hours, the palm oil content in the crystallizer was 12.5 wt%. A sample was taken when the solid fat content as measured by pulse-NMR had reached 23.0 % and this sample was separated in an olein and a stearin fraction using a filter press. The solid fat content of the filter cake, i.e. the stearin fraction, was measured as 66.2 % by pulse-NMR.

A further amount of the mixture of palm olein and palm oil was added to the crystallizer to raise its palm oil content to 13.4 % and the solids content of the crystallizer was allowed to increase to 24.4 wt% when again a sample was subjected to pressure filtration. The solids content of the filter cake was found to be 65.5 wt% as measured by pulse-NMR.

As a control experiment, the crystallizer contents were melted and heated to a temperature of 60 °C, held at this temperature for 1 hour and then cooled to 16 °C. Crystals of good filterability were formed in the early stages of the crystal growth stage but when the solids content in the crystallizer had reached about 18 %, then very many crystals that were quite small and impede filtration started to emerge. A sample taken when the solids content in the crystallizer was 23.3 wt% yielded on pressure filtration a cake with a solids content of only 50.8 %.

The difference in solids content of the filter cake is also reflected in their content of mono-unsaturated triglycerides (MUT) as determined by high pressure liquid chromatography. When using the method according to the invention, the first sample had a MUT-content of 62.7 wt% and the second, slightly less dry sample had a MUT-content of 61.2 wt%. In the reference sample the MUT-content reached only 57.6 wt%.

The example clearly shows that gradual addition of palm oil to palm olein when producing a palm midfraction greatly improves the selectivity of the dry fractionation.

Example 2

In this example, butter fat was dry fractionated and the oil added during the crystal growth phase was the olein fraction resulting from the ongoing fractionation. The same laboratory crystallizer was used as in example 1. About 5.6 kg of butter fat was heated to 60 °C, kept at this temperature for about one hour and then slowly cooled to about 25 °C. At a given solid fat content, a portion of the crystallized fat was subjected to filtration over a laboratory basket centrifuge, the stearin fraction was collected and the olein fraction was recycled into the crystallizer. The fat mixture in the crystallizer was then progressively cooled to 20 °C, 19 °C, 15 °C and 13 °C, and the same procedure was repeated at each of these temperatures. The operation conditions are illustrated in the following table 1.

Table 1

Cycle	1	2	3	4	5
Temperature (°C)	25	20	19	15	13
crystallizer content (kg)	5.60	4.29	4.18	3.92	3.76
SFC prior to filtration (%)	12.9	6.9	6.4	5.3	7.2
Amount filtered (kg)	3.73	0.60	1.52	1.05	3.76
SFC stearin fraction (%)	36.6	36.5	36.5	36.5	36.5
Amount stearin collected (kg)	1.31	0.12	0.27	0.15	0.74
Amount olein recycled (kg)	2.42	0.49	1.25	0.90	3.02

From the above table 1 it appears that an olein fraction is recovered in an amount of 3.02 kg, or 54 wt.%, the remainder being the stearin fractions. The quality of the olein fraction and the stearin fractions is illustrated in table 2 below.

Table 2

Temp. (°C)	Olein	Stea1	Stea2	Stea3	Stea4	Stea5
0	37.3	72.2	73.7	74.4	73.0	77.1
5	26.4	70.8	72.1	72.9	71.3	75.7
10	15.2	66.1	67.7	68.6	64.8	72.1
SFC 15	1.8	56.6	58.0	59.1	56.1	62.6
20	0.0	45.8	47.2	47.9	43.1	50.3
25	0.0	36.8	37.8	39.0	32.7	40.0
30	0.0	26.6	27.3	27.5	21.5	27.7
35	0.0	16.3	16.4	16.7	10.5	15.7

In a comparative experiment, butter fat was heated to about 60 °C, kept at this temperature for about 1 hour and then cooled to 13 °C and allowed to crystallize overnight. The crystallized mass could not be filtered anymore.

Claims

1. Process for dry fractionating oils and fats comprising a nucleation stage, a crystal growth stage and a solids separation stage, wherein at least during the crystal growth stage a triglyceride oil or fat of a composition other than the composition of the oil or fat being dry fractionated is added to the oil or fat being dry fractionated.

2. Process according to claim 1, characterized in that the oil or fat of a composition other than the oil or fat being dry fractionated is added gradually and over an extended period of time.

3. Process according to claim 1, characterized in that the oil or fat of a composition other than the oil or fat being dry fractionated is added continuously.

4. Process according to any of claims 1 to 3, characterized in that the triglyceride oil or fat of a composition other than the oil or fat being dry fractionated comprises triglycerides which easily crystallize out at fractionation temperature.

5. Process according to any of claims 1 to 3, characterized in that the triglyceride oil or fat of a composition other than the oil or fat being dry fractionated is the olein fraction of the ongoing fractionation process or a fraction thereof.

6. Process according to claim 4, characterized in that the oil or fat of a composition other than the oil or fat being dry fractionated is added as a molten liquid oil.

7. Process according to claim 4, characterized in that the oil or fat of a composition other than the oil or fat being dry fractionated is diluted/dissolved with an oil that is substantially liquid at crystallization temperature.

8. Process according to claim 7, characterized in that the oil that is substantially liquid at crystallization temperature is the olein fraction of the ongoing fractionation process or a fraction thereof.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 94 11 6973

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US-A-3 549 386 (JAMES H. MENZIES) * examples I-V * * claim 1 *	1,4,6	C11B7/00
A	EP-A-0 457 401 (UNILEVER) * page 3, line 28 - line 33 * * claims 1,7-12 *	1	
D,A	EP-A-0 399 597 (UNILEVER) * claim 1 *	1	
D,A	EP-A-0 249 282 (UNILEVER) * claims 1,2 *	1	
A	HOFFMANN G. 'The Chemistry and Technology of Edible Oils and Fats and their High Fat Products' 1989 , ACADEMIC PRESS , LONDON, GB * page 253, last paragraph *	1	
A	PATENT ABSTRACTS OF JAPAN vol. 4, no. 42 (C-005) 3 April 1980 & JP-A-55 015 785 (ASAHI DENKA KOGYO KK) 4 February 1980 * abstract *	1	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C11B
A	FR-A-2 455 080 (WALTER RAU LEBENSMITTELWERKE) * example 1 *	1	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	2 February 1995	Dekeirel, M

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
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